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POLAROGRAPHIC DETERMINATION OF IRON AND LEAD IN WASTE OILS

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Quantitative determination of metals introduced into crankcase oil by parts of tractor engines is one of the widely used methods for investigating engine wear.

In several works [1-5], dealing with the determination of metals in waste oils, the iron is separated by extracting the oil with hydrochloric acid or by dry ashing. In both cases the iron is brought into hydrochloric acid solution and then is determined polarographically, in bivalent or trivalent states, using a dropping electrode.

Works of TsNIIAT (Central Scientific-Research Institute of Auto Transport) in 1946-47 [5] demonstrate the considerable advantage of the ashing method. The experience of mass analysis during 4 years corroborated this conclusion, demonstrating accuracy, simplicity, and dependability of the ashing method.

However, in connection with the conversion of motor transport to ethylated gasoline, oils began to reveal lead in quantities many times higher than usual content even after as short a period of engine operation as, for example, 10-20 hours.

During extraction of iron by either method, lead often goes partly into hydrochloric acid solution and partly precipitates as a chloride. Both forms of lead have a negative effect on the accuracy of iron determination, considerably lowering the results of analysis by the usual calculation method utilizing the calibrating curve plotted according to standard solutions. The similar effect of cations and ions on wave heights of lead and arsenic in phosphoric acid solution was studied by T. A. Kryukova [6].

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The purpose of this work is to study the dependence of the polarographic wave of iron solutions on the presence of various quantities of lead iron and lead chloride precipitates.

Experiments were conducted with nitric acid-containing solutions made of twice recrystallized ferrous ammonium sulfate (Mohr's salt) and also of chemically pure metallic iron. The polarograph of the Gor'kiy Institute of Chemistry was used; Model VII, with a mirror galvanometer having an oscillation period of 1.76 seconds, internal resistance 1,115 ohms, critical resistance 12,950 ohms and sensitivity $2.68 \cdot 10^{-9}$ amperes per millimeter per meter. Solutions were heated to 80-90°, cooled to room temperature, and held for polarographic determination in a thermostat at 20°. The quantities of lead chloride introduced into solutions were in the range corresponding to its contents in ashes of waste oil samples for the same volume of hydrochloride solutions.

The sharpest effect of lead ions and precipitates was observed in diluted solutions of iron, e.g., in solutions which are most numerous in production analyses, where the decrease of the wave height reaches 14.8 percent. Deviation from direct proportionality between wave height and concentration decreases with an increase in iron concentration and reaches 5-7 percent for a 0.006-mol solution. Adsorption of iron with lead chloride precipitates is insignificant, being within the limits of measurement errors (1-2 percent). For cases of pure solution of lead chloride without any precipitate, the decrease of the iron wave height amounts to 12 percent for low and 8 percent for medium concentrations.

The dynamics of lead accumulation in ash solutions of oil from a ZIS-120 engine operating on ethylated gasoline shows that saturation of ash solutions with lead chloride is reached after 20 hours of operation. Therefore, the calibrating graph, for oils giving saturated hydrochloride solutions of lead must be plotted according to standard solutions containing lead in quantities corresponding to its complete solubility.

The method suggested for determination of lead consists of wet ashing of oil, conversion of the lead sulfate formed into a soluble state and its polarographic determination on the basis of ammonium acetate. This procedure provides for complete separation of lead from all possible associated metals, as iron, copper, tin, antimony and others.

The method of wet ashing has not been widely used in application to mineral oils because of the long procedure involved (12-15 hours). Decreasing the oil sample to 2 grams as suggested in technical literature /8/ is intolerable because of the low content of lead in waste oil.

The rationalization of the method suggested by the author consists of combined evaporation of oil with its wet ashing. In this way, the entire process of separating the metal from 10 grams of oil may be completed in 5 hours.

The method of extracting metals from oil with a solution of hydrochloric acid, as suggested by the Gor'kiy Institute of Chemistry /9/, also takes 5 hours and the dry ash method with preliminary evaporation of oil takes 7-8 hours.

The calibrating curve for determination of lead concentration has to be plotted according to standard solutions of a chemically pure lead salt using, as an inert electrolyte, an acetate solution of the following concentration: 200 grams of ammonium acetate and 30 milliliters of 80 percent acetic acid per liter of water.

It is also convenient to use, instead of the calibrating line, the mean coefficient indicating the relation between concentrations of standard lead solutions and the heights of polarographic waves relative to these solutions.

Checking this method by introducing into the oil chemically pure metallic lead and also lead chloride with metallic iron, tin, and copper, demonstrated that it gives good results.

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